IS 6716 (1981): Benzoic Acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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IS : 6716 - 1981

Indian Standard

SPECIFICATION FOR BENZOIC ACID, TECHNICAL

(First Revision)

UDC 547·581·2

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1982

AMENDMENT NO. 1 JANUARY 1985

TO

IS:6716-1981 SPECIFICATION FOR BENZOIC ACID, TECHNICAL

(First Revision)

(*Page 8, clause A-7.1, line 2*) - Substitute '60±2°C' *for* '105±2°C'.

(PCDC 9)

Reprography Unit, ISI, New Delhi, India

Indian Standard

SPECIFICATION FOR BENZOIC ACID, TECHNICAL

(First Revision)

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Indian Standard

SPECIFICATION FOR BENZOIC ACID, TECHNICAL

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 5 October 1981, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1972. As a result of the periodical review of the quality of the product available and that specified in Indian as well as overseas standards, the Sectional Committee decided to revise it. In the present revision the requirement for purity of the material has been modified and limits for chlorinated compound, chloride, sulphates, loss on drying and sulphated ash content along with their methods of test have been introduced.

0.3 Benzoic acid is used as preservative for food, fats and in the manufacture of benzoates and benzoyl compounds, dyes; as a mordant in calico printing; for curing tobacco; and as a standard in volumetric analysis and colorimetric analysis. It is also used as an anti-fungal agent in medicine, flavours, perfumes, dentrifices, plasticizers and alkyd resins.

0.4 The food grade benzoic acid is covered under IS : 4448-1967*.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for benzoic acid, technical.

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*Specification for benzoic acid, food grade.

†Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be slightly yellowish to pinkish crystalline powder.

2.2 Solubility — One gram of the material shall be soluble in 3 ml of rectified spirit (*see* IS : 323-1959*).

2.3 The material, when tested according to the methods given in Appendix A, shall also comply with the requirements given in Table 1. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR BENZOIC ACID, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Purity (as C_6H_5COOH) (*on dry basis*), percent by mass, *Min*	99·5	A-2
ii)	Melting point	119 to 121°C	A-3
iii)	Chlorinated compounds	To pass the test	A-4
iv)	Chlorides	do	A-5
v)	Sulphates	do	A-6
vi)	Loss on drying, percent by mass, *Max*	2	A-7
vii)	Sulphated ash, percent by mass, *Max*	0·25	A-8

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in cardboard boxes, bottles, fibre drums, multi-walled paper sacks or as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be marked with the following information:

a) Manufacturer's name and recognized trade-mark, if any;

b) Name of the material;

*Specification for rectified spirit (*revised*).

c) Net mass of the material in the container; and

d) Lot or batch number, in code or otherwise.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.3)

METHODS OF TEST FOR BENZOIC ACID, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF PURITY

A-2.1 Reagents

A-2.1.1 *Phenol Red Solution* — Warm 0·05 g of phenol red [Phenolsulphonphthalein ($C_{19}H_{14}O_5S$)] with 2·85 ml of 0·05 N sodium hydroxide solution and 5 ml of 90 percent ethanol; after solution is effected, add a sufficient quantity of 20 percent ethanol to produce 250 ml.

*Specification for water for general laboratory use (*second revision*).

A-2.1.2 *Standard Sodium Hydroxide Solution* — 0·5 N.

A-2.1.3 *Phenolphthalein Indicator* — Dissolve 0·2 g of phenolphthalein ($C_{20}H_{14}O_4$) in 60 ml of 90 percent ethanol and add a sufficient quantity of water to produce 100 ml.

A-2.2 Procedure — Weigh accurately about 2·5 g of the material and dissolve in 15 ml of warm ethanol previously neutralized using phenol red solution. Add 20 ml of water and titrate with standard sodium hydroxide solution, using phenolphthalein as indicator.

A-2.3 Calculation

$$\text{Purity (as } C_6H_5COOH\text{), percent by mass} = \frac{12{\cdot}212\ VN}{M}$$

where

V = volume in ml of standard sodium hydroxide solution used,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

A-3. DETERMINATION OF MELTING POINT

A-3.1 Procedure — Determine the melting point by the procedure given in IS : 5762-1970*.

A-4. DETERMINATION OF CHLORINATED COMPOUNDS

A-4.1 Apparatus

A-4.1.1 *Silica Crucible*

A-4.1.2 *Nessler Cylinders* — 50 ml capacity, two.

A-4.2 Reagents

A-4.2.1 *Sodium Carbonate A. R.* — Saturated solution in water.

A-4.2.2 *Dilute Nitric Acid* — Mix 190 ml of 50 percent nitric acid with sufficient water to make up to 1 000 ml.

A-4.2.3 *Standard Silver Nitrate Solution* — 0·1 N solution in water.

A-4.2.4 *Standard Hydrochloric Acid* — 0·01 N.

*Methods for determination of melting point and melting range.

A-4.3 Procedure — Weigh out accurately 0·5 g of the material and add to it 10 ml of saturated sodium carbonate solution. Evaporate to dryness and then ignite gently. Take the ignited residue in 20 ml of dilute nitric acid and filter. To the filtrate add 1 ml of standard silver nitrate solution.

A-4.3.1 The material shall be taken to have passed the test if the turbidity obtained shall not be more than that obtained in a similar volume by mixing 1 ml of standard silver nitrate solution and 1 ml of 0·01 N hydrochloric acid.

A-5. DETERMINATION OF CHLORIDES

A-5.1 Apparatus

A-5.1.1 *Nessler Cylinders* — 50 ml capacity, two.

A-5.2 Reagents

A-5.2.1 *Acetone* — *See* IS : 170-1976*.

A-5.2.2 *Standard Silver Nitrate Solution* — 0·1 N.

A-5.2.3 *Standard Hydrochloric Acid Solution* — 0·01 N.

A-5.2.4 *Dilute Nitric Acid* — Mix 190 ml of 50 percent nitric acid with sufficient water to make up to 1 000 ml.

A-5.3 Procedure — Weigh out accurately 1 g of the material and dissolve it in 20 ml of acetone. Add to it 10 ml of water, 10 ml of dilute nitric acid, stir and then add 1 ml of standard silver nitrate solution. Add water to produce 50 ml solution. Any turbidity obtained shall not be more than that obtained in a similar volume by mixing 1 ml of standard silver nitrate solution and 1 ml of 0·01 N hydrochloric acid.

A-6. DETERMINATION OF SULPHATES

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50 ml capacity, two.

A-6.2 Reagents

A-6.2.1 *Acetone* — G. R.

A-6.2.2 *Barium Chloride* — 10 percent solution.

A-6.2.3 *Standard Sulphuric Solution* — 0·01 N.

A-6.2.4 *Dilute Hydrochloric Acid* — 1 : 1.

*Specification for acetone (*second revision*).

A-6.3 Procedure — Dissolve 1 g of accurately weighed material in 20 ml of acetone. Add to this 10 ml of water and 10 ml of barium chloride solution and stir. Further, add 2 ml of dilute hydrochloric acid, and sufficient water to make 50 ml solution. Similarly in another Nessler cylinder take 1·25 ml of standard sulphuric acid, 10 ml of barium chloride solution and 2 ml of dilute hydrochloric acid. Mix the contents.

A-6.3.1 The material shall be taken to have passed the test if the turbidity produced with the material is not greater than that produced with the standard.

A-7. DETERMINATION OF LOSS ON DRYING

A-7.1 Procedure — Weigh accurately about 5 g of the material in a tared porcelain dish and dry the material at 105 ± 2°C. Cool and weigh. Repeat drying, cooling and weighing till two subsequent weighings do not differ by more than 0·001 g.

A-7.2 Calculation

$$\text{Loss on drying, percent by mass} = \frac{(M_1 - M_2) \times 100}{M_1}$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the dried material.

A-8. DETERMINATION OF SULPHATED ASH

A-8.0 Outline of the Method — The material is ignited and burnt until only ash and carbon remain. After cooling, the charred residue is treated with nitric and sulphuric acids and heated until oxidation of the carbon is practically complete. The residue is then cooled and weighed.

A-8.1 Apparatus

A-8.1.1 *Crucible* — squat form of silica, porcelain or platinum.

A-8.1.2 *Bunsen Burner or Muffle Furnace*

A-8.2 Reagents

A-8.2.1 *Concentrated Sulphuric Acid* — relative density 1·84 (*see* IS : 266-1977*).

A-8.2.2 *Nitric Acid* — relative density 1·40 (*see* IS : 264-1976†).

*Specification for sulphuric acid (*second revision*).
†Specification for nitric acid (*second revision*).

A-8.3 Procedure — Weigh accurately about 1 to 2 g of the material in the tared crucible and ignite until thoroughly charred. Cool, moisten the residue with 1 ml of nitric acid and 1 ml of sulphuric acid and cautiously ignite until the carbon is completely consumed. Continue the ignition in a place protected from air currents and using as low a temperature as possible until the combustion of the carbon is complete, cool the crucible in a desiccator and weigh.

A-8.4 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{100\,(M_1 - M_2)}{M}$$

where

M_1 = mass in g of the ignited residue with the crucible,

M_2 = mass in g of the crucible, and

M = mass in g of the material taken for the test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF BENZOIC ACID, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry and air-tight glass containers or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, batch number and other important particulars of the consignment (*see* **4.1**).

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SAMPLING INSTRUMENT

B-2.1 The sampling instrument is a closed type sampling tube, undivided (*see* Fig. 1). It consists of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel), one closely fitting into the other throughout their length so that it is possible to rotate one tube within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one third the circumference are cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sampler is usually provided with a locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm, proportionately to the length. A length of 150 cm and a diameter of 30 mm can cater for most needs.

B-2.1.1 *Use of Sampling Instrument* — The instrument is inserted in closed position in an oblique direction till it touches the bottom. The material is admitted by rotating and opening the tubes and finally closing them, withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'cores' shall be taken from different parts of the same container such that they are at least 75 mm in the case of drums, bags, etc, and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches the bottom so that an entire cross section of the material is withdrawn.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In a single consignment, all the containers of the same size, containing material from the same batch of manufacture, shall constitute a lot.

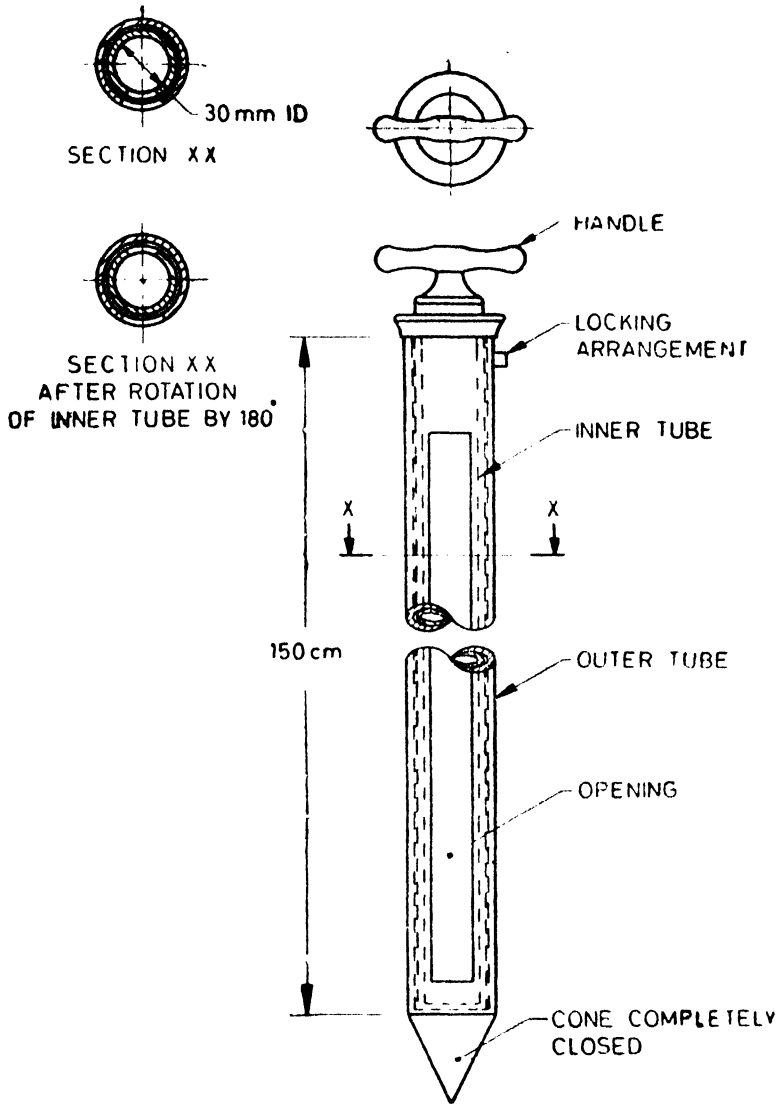

FIG. 1 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

B-3.2 For ascertaining the conformity of the material to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected from a lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Number of Containers in the Lot (N)	Number of Containers to be Selected (n)
(1)	(2)
4 to 25	3
26 " 50	4
51 " 100	5
101 " 150	6
151 " 300	7
301 and above	8

NOTE — When the size of the lot is three or less, all the containers shall be sampled.

B-3.3 These containers shall be chosen at random from the lot and in order to ensure randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a random table is not available, the following procedure shall be adopted:

Starting from any container count them in one order as 1, 2, 3,....., up to r and so on, where r is the integral part of N/n (N being the number of containers in the lot and n the number of containers to be selected). Every rth container thus counted shall be withdrawn to constitute the sample.

B-4. TEST SAMPLES AND REFEREE SAMPLE

B-4.1 From each of the containers selected according to **B-3.2**, a representative portion of the material shall be drawn. These samples shall constitute individual samples.

B-4.2 From each of these individual samples (**B-4.1**), an equal quantity of the material shall be drawn and thoroughly mixed to constitute a composite sample not less than 600 g. The composite sample shall be divided into three equal parts and packed in clean bottles and labelled with full identification particulars of the samples, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-5. TESTS

B-5.1 Tests for the determination of all the characteristics of this specification shall be carried out on the composite samples.

B-6. CRITERIA FOR CONFORMITY

B-6.1 For determining the conformity of the lot to this specification, the test results on the composite samples shall meet the corresponding requirements specified in this standard.

(*Continued from page* 1)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg m/s^2
Energy	joule	J	1 J = 1 N m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 26 60 21, 27 01 31 **Telegrams : Manaksanstha**

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